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(21) International Application Number: PCT/ZA00/00019 (22) International Filing Date: 8 February 2000 (08.02.00) (30) Priority Data: 99/0969 8 February 1999 (08.02.99) ZA (71) Applicant (for all designated States except US): SAPPI LIMITED [ZA/ZA]; 6th Floor, Sappi House, 48 Ameshof Street, Braamfontein, 2017 Johannesburg (ZA). (72) Inventor; and (75) Inventor/Applicant (for US only): MABOKA, Sydney [ZA/ZA]; 20 Cathedral Street, 6529 George (ZA). (74) Agent: D.M. KISCH INC.; P.O. Box 781218, 2146 Sandton (ZA).		(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: TIMBER PROCESS AND PRODUCT (57) Abstract This invention provides a binding mixture for use in manufacturing chipboard, the mixture including an effective amount of methyl di-isocyanate ("MDI") and one or more polyurethane catalyst(s) being selected from the following: (a) one or more amine compound(s) including aliphatic and aromatic tertiary amine derivatives of phenols, esters, ethers, alkenes and/or alcohols; or (b) one or more organometallic compounds of tin, bismuth, zinc, iron, and/or alkali metal salt(s); or (c) suitable mixtures of (a) and (b) above.		

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TIMBER PROCESS AND PRODUCT**5 INTRODUCTION**

This invention relates to a timber process and product. More particularly this invention relates to a process for manufacturing so-called particle board or chipboard, and to a timber product in the form of such particle
10 board or chipboard when manufactured by or from the aforementioned process.

BACKGROUND TO THE INVENTION

15 It is known in the manufacture of particle board or chipboard (hereinafter referred to as "chipboard") that a urea-formaldehyde resin (hereinafter referred to as "UF") is used as a binder or adhesive to bond the particles or chips of wood.

20 Typically such binders contain approximately 55% to 60% formaldehyde (in moles) but because of recent awareness of the health problems associated with the use of formaldehyde, the amount of formaldehyde in such resins is generally being reduced. Hence the amount of formaldehyde in the aforementioned resins has been reduced to
25 approximately 50% or less (in moles).

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The aforementioned resins need to be cured, and the curing process is accelerated by heating in a press under pressure i.e. heat is applied by hot metal platens on both sides of a so-called mat of glued chips. Such chipboard pressing takes place either in a batch-type (using daylight presses) or in a continuous process i.e. using continuous (for example roller) presses.

It is also known in the chipboard industry that methyl di-isocyanate (hereinafter referred to as "MDI") may be added to the aforementioned type of resin to hasten the curing process i.e. to effectively increase the speed of the curing process and hence to reduce the curing time. However, because of the high cost of MDI and the amount required to effectively hasten the curing process, the use of MDI is not cost-effective on an industrial scale.

OBJECTS OF THE INVENTION

It is accordingly a general object of the present invention to provide an improved binding mixture for chipboard manufacture.

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It is also an object of the present invention to provide an improved process in which such improved mixture is used, which is both cost-effective and which increases the efficiency and rate of curing of the binding mixture.

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It is a further object of the invention that the aforementioned binding mixture and process will lead to an increase in productivity or production flowing from shorter curing periods resulting from use of the aforementioned binding mixture.

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SUMMARY OF THE INVENTION

According to one aspect of the present invention, there is provided a binding mixture for use in manufacturing chipboard, the mixture including
15 an effective amount of MDI and one or more polyurethane catalyst(s) being selected from the following:

- (a) one or more amine compound(s) including aliphatic and aromatic tertiary amine derivatives of phenols, esters, ethers, alkenes and/or alcohols; or
- 20 (b) one or more organometallic compound(s) of tin, bismuth, zinc, iron and/or alkali metal salt(s); or
- (c) suitable mixtures of (a) and (b) above.

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The MDI may or may not be water-emulsifiable, and the or each polyurethane catalyst(s) may or may not be (a) delayed-action catalyst(s).

- 5 To the mixture may be added an effective amount of toluene diisocyanate ("TDI") and/or one or more internal wetting and release agents.

The aforementioned mixture(s) may of course include a suitable amount
10 of urea-formaldehyde resin which may or may not have suitable quantities of polyol(s) added thereto.

The amount of formaldehyde in the aforementioned urea-formaldehyde resin may preferably be approximately 50% (in moles) i.e. may be less
15 than, equal to, or more than 50% (in moles).

By using a catalyst as aforesaid, the effective amount of MDI required is reduced relative to the amounts known in the prior art to be sufficient to hasten the curing process of the mixture.

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Suitable amines or amine compounds may be selected from the following:

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2-dimethyl ethanolamine (hereinafter referred to as "DMEA");
di-amino bicyclo-octane (hereinafter referred to as "DABCO"); and
N,N-dimethyl cyclohexylamine (hereinafter referred to as "DMCHA").

- 5 Suitable organometallic tin-based compounds may be selected from the following:

stannous octoate, dibutyl tin dilaurate, dibutyl tin mercaptide, dibutyl tin thiocarboxylate, and dioctyl tin thiocarboxylate.

- 10 Other suitable organometallic compounds may include ferric acetylacetonate.

Suitable alkali metal salts may be selected from the following:
calcium carbonate, salts of carbonic acid, and salts of acetic acid.

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One supplier of the above catalysts (as mixtures or single compounds) is Air Products, South Africa, and the catalysts may be identified inter alia by the following trade marks/brands and/or acronyms, respectively:

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<u>CHEMICAL BRAND NAME</u>	<u>ACTIVE INGREDIENT(S)</u>
DMEA	2-Dimethyl ethanolamine
DABCO	Di-amino bicyclo-octane
POLYCAT 8 (DMCIA)	<u>N,N-dimethyl cyclohexylamine</u>
DABCO R-8020	Triethylenediamine and Dimethylethanolamine
DABCO DC-1	Tin and amine complexes
DABCO DC-2	Tin and amine complexes
DABCO K-15	Potassium octate and diethylene glycol
DABCO TMR 2	Quaternary ammonium salt in ethylene glycol
THORCAT 401	Di-N-butyltindilaurate

When used with one or more of the above catalysts, the amount of MDI required to effect a suitable hastening of the curing process may preferably be in the range of about 0.1% to 1.9% on bone dry wood (hereinafter referred to as "BDW").

According to another aspect of the present invention, there is provided an additive for a binding mixture for use in manufacturing chipboard, the additive including an effective amount of MDI and a catalyst, each as indicated above, and as otherwise described herein.

According to a further aspect of the present invention, there is provided a process for manufacturing chipboard, the process including the steps of

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adding to or using in a binding mixture consisting of urea-formaldehyde resin, an effective amount of MDI and a catalyst, each catalyst being as indicated above, or as otherwise herein described, or an additive, respectively, as herein described, and suitably mixing the binding mixture
5 with wood particles and/or chips.

DETAILED DESCRIPTION OF THE INVENTION

The invention will now be described in greater detail by way of non-
10 limiting example(s), with reference to the following:

1. LABORATORY TESTS

Laboratory tests were carried out by way of reasonable technical trials,
15 by personnel of the applicant's wholly owned subsidiary namely Sappi Timber Industries (Proprietary) Limited on wood particles and chips as used for manufacturing chipboard in the factories of the aforementioned company.

20 Using the aforementioned raw materials, binder mixtures of urea-formaldehyde resin as typically used in the aforementioned company's production facilities were applied to the aforementioned particle chips,

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and compared with urea-formaldehyde mixtures including MDI, and further compared with mixtures of urea-formaldehyde with MDI and various catalysts as set out above.

5 The resulting mixtures of particles/chips and binder mixtures were pressed in a laboratory scale press to simulate plant production conditions, and curing times were measured. In the binding mixtures that did not include a catalyst, longer curing times were observed.

10 Details of the mixtures used and the corresponding results are set out in Table 1 hereafter:

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TABLE 1

MIXTURE ADDED TO UF RESIN	INCREASE IN PRODUCTION SPEED (%)
<u>MDI + RELEASE AGENT</u> 1.4 % (on BDW) of MDI + 0.5 % (on UF solids) Internal Release and Wetting Agent	 34.6
<u>MODIFIED MDI</u> (a) 1.4% (on BDW) MDI modified with PPG diol MDI pre-polymer (b) 1.4% (on BDW) MDI modified with PPG Triol MDI pre-polymer (c) 1.4% (on BDW) MDI modified with TDI (d) 1.4% (on BDW) MDI modified with PPG Triol TDI pre-polymer	 44.3 44.3 44.3 42.4

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Table 1 (Contd)

<u>CATALYSTS</u>		
(a)	1.4% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% DABCO TMR 2 catalyst	45.8
(b)	1.4% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UF mass) DABCO K 15 catalyst	44.3
(c)	1.4% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UFmass) DABCO DC 2 catalyst	44.3
(d)	1.2% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UFmass) DMCHA catalyst	42.3
(e)	(1.2% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UF mass) DABCO R8020 catalyst	42.3
(f)	1.2% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UF mass) Thorcat catalyst	40.0
(g)	1.4% (on BDW) MDI modified with Polyethylene glycol MDI pre-polymer + 0.1% (on UF mass) DABCO DC 2 catalyst	42.8
(h)	0.5% (on BDW) MDI + 1.1% (on UF mass) DMEA catalyst	33.3
(i)	0.5% (on BDW) water emulsified MDI + 1.1% (on UF mass) DMEA catalyst	33.3

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In the aforementioned table, the brand name Daltolac R130 is a brand name for polyether polyol, which is sold by ICI (Imperial Chemical Industries). Similarly PPG diol MDI Pre-Polymer is the brand name for polypropylene glycol diol MDI pre-polymer and PPG triol TDI Pre-Polymer is the brand name for polypropylene glycol triol TDI pre-polymer.

The curing times for mixtures where typically 0.1% to 1.9% MDI and one or more of the abovementioned catalysts were added (on BDW), were the shortest.

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The results of the above tests indicated that in all cases the binding mixtures with MDI and one or more catalysts resulted in a shorter curing period with the same or an increased binding strength.

15 It was also found that the use of amines as catalysts alone resulted in the least costly mixture and hence the most cost-effective process(es).

2. PRODUCTION-PLANT TRIALS

20 The aforementioned tests were repeated by way of reasonable technical trials, on two different production lines namely one using a single daylight press and the other using a continuous press. Details of the

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mixtures used and the corresponding results are set out in Table 2 hereunder:

TABLE 2

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MIXTURE ADDED TO UF RESIN	INCREASE IN PRODUCTION SPEED (%)
<u>CONTINUOUS PRESS</u> <u>WHITE RIVER FACTORY</u> 0.5% (on BDW) waster emulsifiable MDI + 1.1% (on UF mass) + DMEA catalyst	14.3

The abovementioned production-line tests confirmed the aforementioned laboratory results, with improvements in or increased curing times of up to approximately 14 to 24%. The applicant believes that it may be possible to achieve greater improvements than the aforementioned.

It will therefore be seen from the aforementioned that a considerable improvement in curing times and hence in productivity may be possible by using the aforementioned invention. Such improvement also appears to be cost-effective relative to increased productivity and production on an industrial scale.

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Although certain embodiments only of the invention have been described herein, it will be apparent to any person skilled in the art that other variations and/or modifications of the invention are possible. Such variations and/or modifications are therefore to be considered as falling
5 within the spirit and scope of the invention as claimed hereinafter.

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CLAIMS

1. A binding mixture for use in manufacturing chipboard, the mixture
5 including an effective amount of methyl di-isocyanate ("MDI") and
one or more polyurethane catalyst(s) being selected from the
following:
 - (a) one or more amine compound(s) including aliphatic and
aromatic tertiary amine derivatives of phenols, esters,
10 ethers, alkenes and/or alcohols; or
 - (b) one or more organometallic compounds of tin, bismuth,
zinc, iron, and/or alkali metal salt(s); or
 - (c) suitable mixtures of (a) and (b) above.
- 15 2. A binding mixture as claimed in claim 1, wherein the MDI is water-
emulsifiable.
3. A binding mixture as claimed in either claim 1 or claim 2, wherein
the or each polyurethane catalyst(s) is/are (a) delayed-action
20 catalyst(s).
4. A binding mixture as claimed in any one of the preceding claims,
wherein an effective amount of toluene di-isocyanate ("TDI") is
added to the MDI.

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5. A binding mixture as claimed in any one of the preceding claims, wherein one or more internal wetting and release agents is/are added to the mixture.

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6. A binding mixture as claimed in any one of the preceding claims, wherein a suitable amount of urea-formaldehyde resin is added.

10

7. A binding mixture as claimed in claim 6, wherein a suitable quantity of or more one polyol(s) is/are added.

8. A binding mixture as claimed in either claim 6 or claim 7, wherein the amount of formaldehyde in the urea-formaldehyde resin may be approximately 50% (in moles).

15

9. A binding mixture as claimed in any one of the preceding claims, wherein the or each suitable amine(s) or amine compound(s) are selected from the following:

2-dimethyl ethanolamine ("DMEA");

20

di-amino bicyclo-octane ("DABCO"); and

N,N-dimethyl cyclohexylamine ("DMCHA").

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10. A binding mixture as claimed in any one of the preceding claims, wherein the or each suitable organometallic tin-based compound(s) is/are selected from the following:

Stannous octoate, dibutyl tin dilaurate, dibutyl tin mercaptide,
5 dibutyl tin thiocarboxylate, and dioctyl tin thiocarboxylate.

11. A binding mixture as claimed in any one of the preceding claims, wherein the suitable organometallic compounds include ferric acetylacetonate.

10

12. A binding mixture as claimed in any one of the preceding claims, wherein the suitable alkali metal salts are selected from the following:

calcium carbonate, salts of carbonic acid, and salts of acetic acid.

15

13. A binding mixture as claimed in any one of the preceding claims, wherein the amount of MDI required to effect a suitable hastening of the curing process is in the range of from about 0.1% to 1.9% of bone dry wood ("BDW").

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14. A binding mixture substantially as herein described and/or exemplified.

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15. An additive for a binding mixture for use in manufacturing chipboard, the additive including an effective amount of MDI and a catalyst, each catalyst being as claimed in any one of the preceding claims.

5

16. An additive for a binding mixture for use in making chipboard, substantially as herein described and/or exemplified.

10

17. A process for manufacturing chipboard, the process including the steps of adding to or using in a binding mixture an effective amount of MDI and a catalyst, each catalyst being as claimed in any one of claims 1 to 14, and suitably mixing the binding mixture with wood particles and/or chips.

15

18. A process for manufacturing chipboard, the process including the steps of adding to or using in a binding mixture an effective amount of an additive as claimed in either claim 15 or claim 16, and suitably mixing the binding mixture with wood particles and/or chips.

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19. A process for manufacturing chipboard, substantially as herein described and/or exemplified.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
 IPC 7 C08L97/02 C08G18/16

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IPC 7 C08L C08G

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

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